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RESEARCH MEMORANDUM

THERMAL SHOCK RESISTANCE AND HIGH-TEMPERATURE STRENGTH OF A MOLYBDENUM DISILICIDE - ALUMINUM OXIDE CERAMIC

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SUMMARY

A ceramic consisting nominally of 75 percent molybdenum disilicide and 25 percent aluminum oxide was investigated to determine its thermal shock characteristics and high-temperature strength properties.

In a rim-quench thermal shock evaluation, the material was found to be superior to pure molybdenum disilicide. In a simulated altitude blow-out test, the material withstood an average of $2\frac{1}{2}$ cycles when quenched from 1800° F. The modulus-of-rupture strengths varied from 25,100 pounds per square inch at 1800° F to 12,000 pounds per square inch at 2200° F.

INTRODUCTION

Molybdenum disilicide possesses outstanding high-temperature strength and oxidation resistance (ref. 1); however, utilization of the material is limited by its low thermal-shock resistance. Molybdenum disilicide is also known to possess the ability to bond such oxides as alumina and zirconia, and the strength obtainable from such a mixture as well as the thermal shock behavior is therefore of interest. Specimens of molybdenum disilicide containing approximately 25 percent of aluminum oxide were evaluated for thermal-shock characteristics. Two tests were used, one simulating altitude blow-out conditions in a jet engine and the other a "rim-quench" method. Modulus-of-rupture strengths were also determined at temperatures to 2200° F and a flexure-creep evaluation was made at 2000° F.

All specimens were prepared by the P. R. Mallory Company of Indianapolis, Indiana and are of the composition designated D-1922 by the manufacturer. The evaluations described were carried out at the NACA Lewis laboratory.

APPARATUS AND PROCEDURE

All specimens were tested in the as-received condition after radiographic inspection, density, and hardness determinations. Chemical analyses and metallographic examinations were made on broken fragments.

Thermal-shock evaluation. - For the rim-quench test, the apparatus consists of a vertical electrical resistance furnace from which the specimen can be rapidly lowered into a tank of water of known temperature. To assure the loss of heat from the rim only, the disk specimens (2 in. in diam. and 1/4 in. thick) are held between two low-conductivity alloy cylinders with asbestos washers between the specimen and the cylinders. The entire specimen assembly is heated and quenched as a unit. Figure 1 pictures the furnace, quench tank, and specimen-holder assembly for the rim-quench thermal-shock apparatus.

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To perform a test in the rim-quenching apparatus, a furnace temperature is selected to give a quench of less severity than is believed necessary to cause failure. The specimen is placed in the furnace at the selected temperature, and as soon as a thermocouple in contact with the specimen indicates that temperature equilibrium is attained, the specimen assembly is rapidly quenched by lowering it into the water. After cooling, nonporous specimens are examined for cracks by a penetrant oil method. If no crack is found, the test is repeated using increasingly higher furnace temperatures until a temperature difference that causes failure is determined.

The apparatus for the simulated altitude blow-out test consists of a furnace in which the specimen is heated and an adjacent air-duct into which the specimen may be quickly withdrawn from the furnace. A more complete description of the apparatus is given in reference 2. The quenching air flow through the duct is 50 pounds per minute (265 ft/sec) at 70° to 80° F. In conducting a thermal-shock evaluation, 25 quench cycles are made from a temperature of 1800° F, and if the specimen survives, the temperature is increased to 2000° F for another 25 cycles and so on with 25 cycles each at 2200° and 2400° F or until failure by cracking occurs.

Modulus-of-rupture evaluation. - Specimens 1/2 by 1/4 by $2\frac{1}{2}$ inches in size were employed. The test consists of supporting the specimens on two knife edges $1\frac{1}{2}$ inches apart with a loading knife edge bearing on the center of the upper surface. Load is applied until failure occurs, and the modulus of rupture strength is calculated from the equation:

$$\text{Modulus of rupture strength, lb/sq in.} = \frac{3/2\ pd}{wt^2}$$

where

p = load on specimen at failure, lb

d = distance between supporting knife edges, in.

w = specimen width, in.

t = specimen thickness, in.

For the room-temperature evaluations, a conventional tensile machine was employed to apply load to the specimen. For the high-temperature determinations, the specimens were placed on silicon carbide knife edges mounted in a furnace. The loading knife edge is part of a plunger connected to a lever system outside the furnace. The load is applied by running a controlled flow of water to a receptacle connected to the lever. The water flow was adjusted to produce a rate of loading of 2000 pounds per square inch per minute.

Flexure-creep evaluation. - Flexure-creep evaluation is a long-time strength evaluation in which the specimen is stressed in bending instead of the more conventional axial tension. The specimen is placed on the knife edges in the furnace at temperature as in the modulus-of-rupture test and a load sufficient to produce the desired stress is applied and maintained until failure occurs. Failure times are determined, or in the case of materials in which creep is an important factor, creep rates may be measured.

RESULTS AND DISCUSSION

Inspection and Analysis

Radiographic inspection showed segregation and porosity in all but one of the thermal shock disks. The modulus-of-rupture bars, while more free of voids, showed some segregation. Density determinations by the water-immersion method also disclosed considerable porosity. The average of the water-immersion determinations was 4.66 grams per milliliter. Densities determined by weighing and calculating volumes from micrometer measurements gave an average of 4.1 grams per milliliter. Considerable porosity was also revealed by metallographic examination. The microstructure also indicated that the bonding between the alumina and the molybdenum disilicide appeared to be good. Hardnesses taken on the polished surfaces varied between 69 and 72 and averaged at 71.1 Rockwell superficial 30-N.

Samples prepared to represent one-half of the modulus-of-rupture bars were analyzed chemically with the following results:

Element	Analyst A, percent by weight	Analyst B, percent by weight
Mo	47.2	47.1
Si	26.7	26.7
Al_2O_3	25.7	----
Fe	----	0.5
Ca	----	0.2 ± 0.1

<u>Thermal-shock evaluation</u>. - The results of the rim-quenching evaluations are given in table I and compared with results for molybdenum disilicide and a titanium carbide - 20 percent cobalt cermet. While higher than those for pure molybdenum disilicide, the temperature differences survived by the alumina plus molybdenum disilicide specimen were considerably below those causing failure in the cermet. This relation holds also for simulated blow-out evaluation. As shown on table II, the alumina plus molybdenum-disilicide specimen withstood an average of $2\frac{1}{2}$ cycles from 1800° F which can be compared with molybdenum disilicide at 2 cycles and with the cermet which did not fail after a total of 100 cycles, 25 each at 1800°, 2000°, 2200°, and 2400° F. The cermet is considered to have adequate thermal-shock resistance for turbine blade use (ref. 3). 2962

The air-quench test has been devised primarily for evaluating the relative merits of materials under simulated blow-out conditions and while survival of 100 cycles is considered to be adequate, the minimum number of cycles to be survived to insure suitability for a turbine blade application has not been determined. The alumina plus molybdenum disilicide material is inferior to the cermet and a life of only $2\frac{1}{2}$ cycles would not appear promising, but an actual blade operation test would be required to give a definite answer as to the suitability of the material for turbine blade use.

That there has been a large improvement in thermal-shock resistance over that of pure molybdenum disilicide is shown by the rim-quench test listed in table I, and the material may be of value for applications involving moderate thermal-shock conditions.

<u>Modulus-of-rupture evaluations</u>. - Modulus-of-rupture strengths at four temperatures are listed in table III. A sharp decrease in strength is observed above 2000° F. While these short time strengths are less than for the pure molybdenum disilicide, as shown in table III, they are approaching the strength of the cermet at the higher temperatures. The material failed in a brittle manner without apparent deformation in all of the modulus-of-rupture tests.

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Flexure-creep evaluation. - The flexure-creep specimens listed in table IV also failed in a brittle manner. With pure molybdenum disilicide, 1 percent elongation took place in a 24-hour period at 30,100 pounds per square inch stress in a similar test at the same temperature (ref. 4). Observations of failed specimens after these and other high-temperature tests indicates that the alumina plus molybdenum-disilicide mixture tends to maintain the high oxidation resistance of pure molybdenum disilicide.

SUMMARY OF RESULTS

A ceramic consisting of 75 percent molybdenum disilicide and 25 percent aluminum oxide was investigated with the following results:

1. In a rim-quench thermal-shock evaluation, the material was found to be superior to pure molybdenum disilicide. In a simulated altitude blow-out test, the material withstood an average of $2\frac{1}{2}$ cycles when quenched from 1800° F.

2. The modulus-of-rupture strengths were found to vary from 25,100 pounds per square inch at 1800° F to 12,000 pounds per square inch at 2200° F.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
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TABLE I. - THERMAL-SHOCK BEHAVIOR OF $MoSi_2$-Al_2O_3 SPECIMENS

IN RIM-QUENCHING EVALUATION

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Specimen	Temperature difference between specimen and water bath at start of quench, °F					
	$MoSi_2$-Al_2O_3		$MoSi_2$		Cermet[a]	
	Survived	Failed	Survived	Failed	Survived	Failed
1	700	800	325	350	1600	1700
2	600	700	325	350		
3	500	600	250	300		

[a]80 Percent TiC plus 20 percent cobalt by weight.

TABLE II. - SIMULATED ALTITUDE BLOW-OUT TEST FOR $MoSi_2$ PLUS Al_2O_3

Specimen number	Furnace temperature, °F	Number of cycles survived
4	1800	$2\frac{1}{2}$
5	1800	3
6	1800	2

TABLE III. - MODULUS OF RUPTURE STRENGTH OF $MoSi_2$-Al_2O_3 SPECIMENS

Temperature, °F	Modulus of rupture strength, psi		
	$MoSi_2$-Al_2O_3	$MoSi_2$[a]	Titanium carbide cermet[b]
Room	23,300		
	22,100		
1800	24,500	68,500	59,200
	25,100	66,000	58,150
2000	22,600	84,700	35,850
	23,200	87,800	35,400
2200	11,300	53,000	18,750
	12,000	56,000	17,700

[a]Ref. 4.

[b]Ref. 5.

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TABLE IV. - BEHAVIOR OF $MoSi_2-Al_2O_3$ SPECIMENS

IN FLEXURE-CREEP TEST

Specimen number	Temperature, °F	Stress, psi	Life	
			hr	min
1	2000	15,000	2	7
2	2000	15,000	2	26

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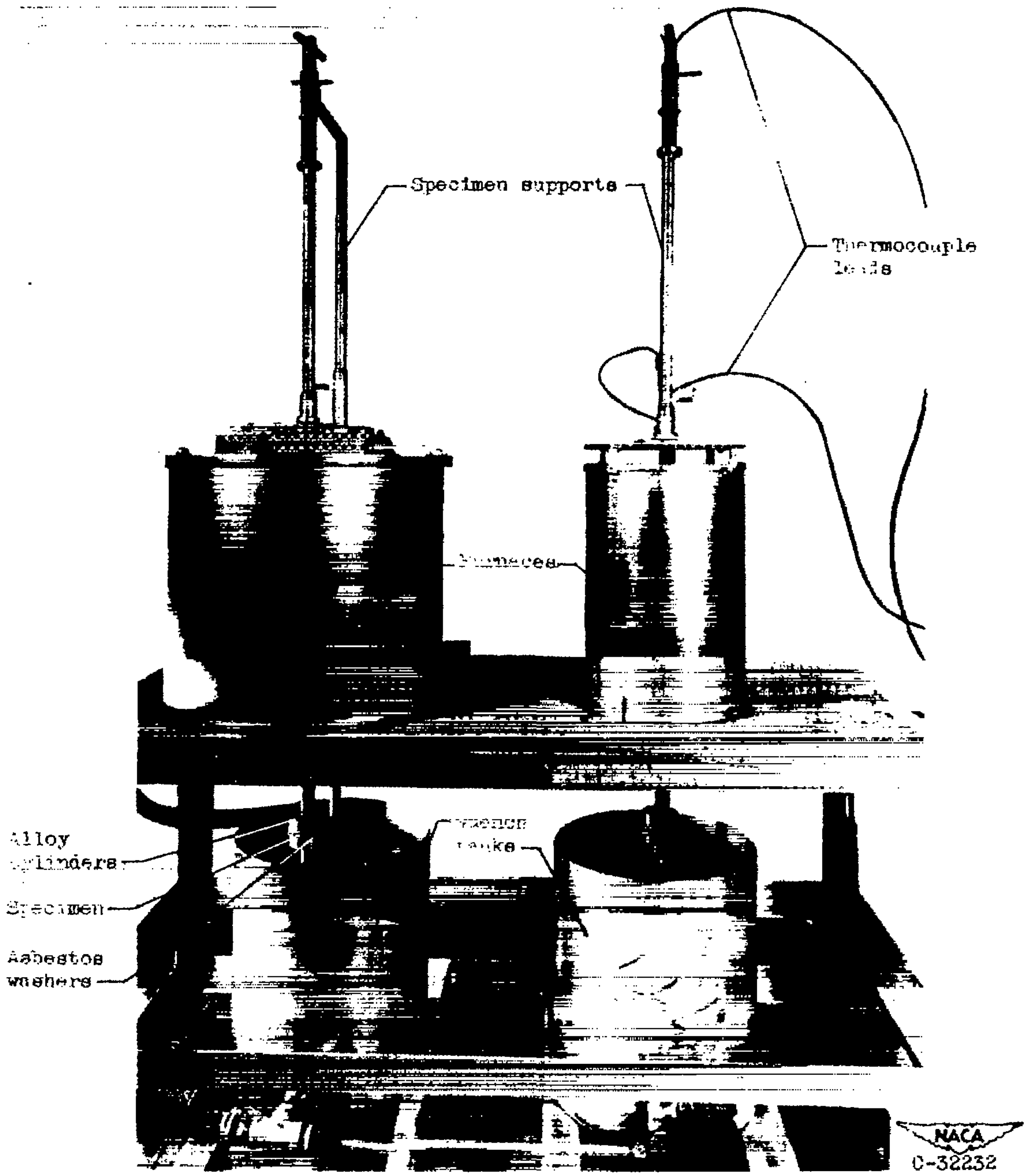

Figure 1. - Rim-quench thermal-shock evaluation apparatus. Globar furnace for high-temperature quenching at left with wire-wound furnace for moderate temperatures at right.

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